THE BOEING COMPANY Space Division

IDENTIFYING OPTIMUM PARAMETERS OF HOT EXTRUSIONS

Contract NAS 7-276

Prepared for

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WORK IN PROGRESS

Extrusion

Furnace Preparation

A new furnace for heating billets for extrusion is being constructed. The coil will be lined with ZrO₂ and coupled directly to the metal can. This construction is similar to the furnace originally used. The originally available ZrO₂ lined furnace was abandoned because it was not large enough for many of the billets. Use of the available graphite susceptor (carbon black insulated) furnace recently used for extrusion is being discontinued due to recent carbon-metal eutectic melting problems.

Extrusion Preparation

Billets for four extrusions have been sent to Nuclear Metals where cans and dies are being prepared during the above furnace construction. These are:

- 1. 1.0 and 1.5-inch diameter Al_2O_3 billets to be extruded in a TZM can.
- 1.0 and 1.5-inch diameter MgO + 5 w/o Al₂O₃, MgO + 1/2 w/o ZrO₂, MgO + 1 w/o CaO, and MgO + 2 w/o CaO billets to be extruded in a tungsten can.
- 3. MgO and a few MgO alloy billets with square cross sections between about .6" and 1.0" on a side to be extruded in a tungsten can with a round O.D. through a round die. These include single crystals with approximate < 100 >, < 110 > and < 111 > axial orientations to check the effect of starting orientation on resultant extruded texture. The square shaped polycrystalline billets are intended to investigate causes of cracking, and possibly allow cutting of specimens with higher index textures along the tensile axis. Such higher index specimens are expected to be substantially stronger.
- 4. Cylindrical MgO billets 0.75" to 2.0" in diameter to be extruded in a round tungsten can through a square or rectangular die. This is another approach to obtaining specimens with textures of higher index than the normal 100 texture.

Fracture Analysis

Further fracture analysis of extruded specimens is continuing. Figure 1 is a composite photograph of a complex fracture originating from one (or more) grain boundary surfaces approximately parallel to the fracture surface (Type 1 fracture). Of particular interest is the grain boundary surface nearest the specimen tensile surface which has faint off-sets highly suggestive of slip bands, apparent chipping and several close slip bands. This is rather suggestive of the mechanism of edge band fracture in bi-crystals shown by Stokes and Li(1).

As previously noted (2), one of the possible reasons fracture origins in MgO bi-crystals may not have been noted is due to orientation. Previous tests have used bi-crystals with the boundary perpendicular to the tensile axis so edge bands will normally reach it first and cause failure. Further, screw bands that did meet the boundary would be less likely to cause fracture due to the higher stresses apparently required and to the low angle between the screw band and the boundary. The latter could be significant since, as the boundary-screw band angle approaches zero, the screw band may see the least difference between tilt boundaries and the adjoining grains.

Therefore, some cursory room temperature bi-crystal flexure tests with the bi-crystal boundaries parallel to the tensile axis were made. Specimens were annealed at 3600°F(2035°C), chemically polished, then tested. These all failed by cleavage as. expected, even when the cleavage angle to the tensile axis was high (see Figures 2-4). Figure 3 shows the tensile surface of a bi-crystal (Mb-3-1) which more nearly approached the conditions surrounding longitudinal boundaries in extruded MgO. This shows that both edge and screw bands were stopped at the boundary. Examination of the fracture surface (Figure 4) suggests an interior origin with several screw bands near the area of probable origin. However, it is also possible that fracture started at the boundary on the surface from either of the stopped screw or edge bands there, then propagated down the boundary a distance before branching out by cleavage into the two grains.

FUTURE WORK

The four extrusions now waiting completion will be extruded shortly. Further extrusion efforts will concentrate on reducing grain size and changing texture (for higher strength). Some of these extrusions will also be carried on to determine the origin of cracking in extrusions and for further examination of other oxides and alloys.

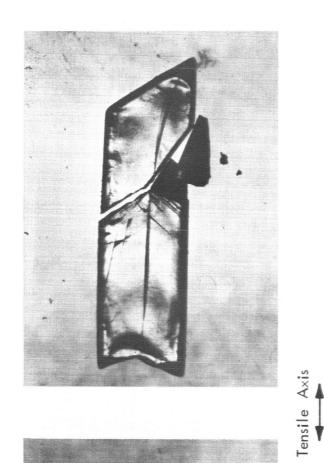
High temperature testing of MgO will be conducted since the improvement in properties from hot extrusion is expected to be as much or more than at room temperature. Further work on fracture mechanisms will be conducted as warranted.

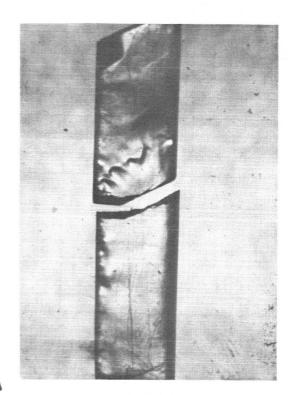
REFERENCES

- R. J. Stokes, C. H. Li, "Dislocations and the Strength of Polycrystalline Ceramics", pp. 133–158 in Materials Science Research, Vol 1 (ed. by Stadelmaier and Austin), Plenum Press, New York (1963).
- 2. R. W. Rice, J. G. Hunt, "Identifying Optimum Parameters of Hot Extrusions", Interim Report II, NASA Contract NAS 7-276, May 1966.



FIGURE 1 - FRACTURE ORIGIN AREA OF SPECIMEN M-f-5 $_{1-1}$







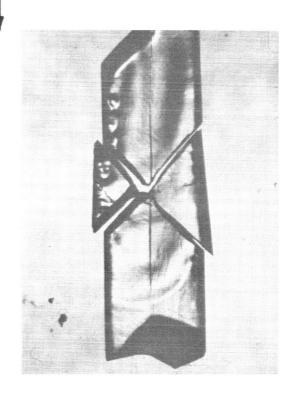
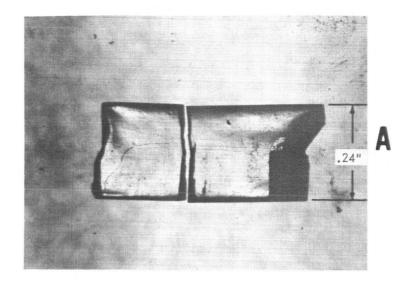


FIGURE 2 - FAILURE OF MgO BI-CRYSTALS OF VARIOUS ORIENTATION WITH THE BOUNDARY PARALLEL TO THE TENSILE AXIS



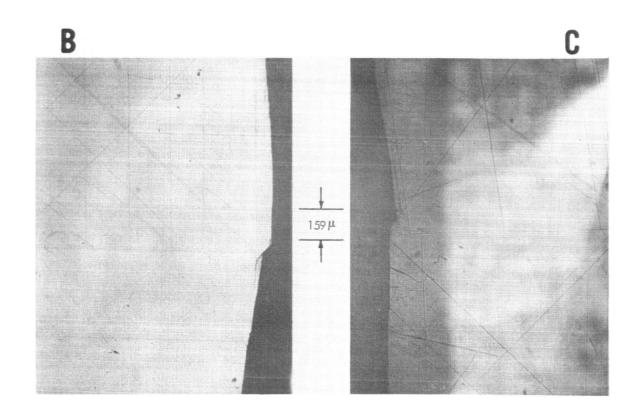


FIGURE 3 - TENSILE SURFACE OF MgO Bi-CRYSTAL Mb-3-1 WITH ABOUT 15° TILT PARALLEL TO THE TENSILE AXIS (A) Macro Photo (B) & (C) Micro Photo of Matching Halves (etched-edge bands are diagonal)

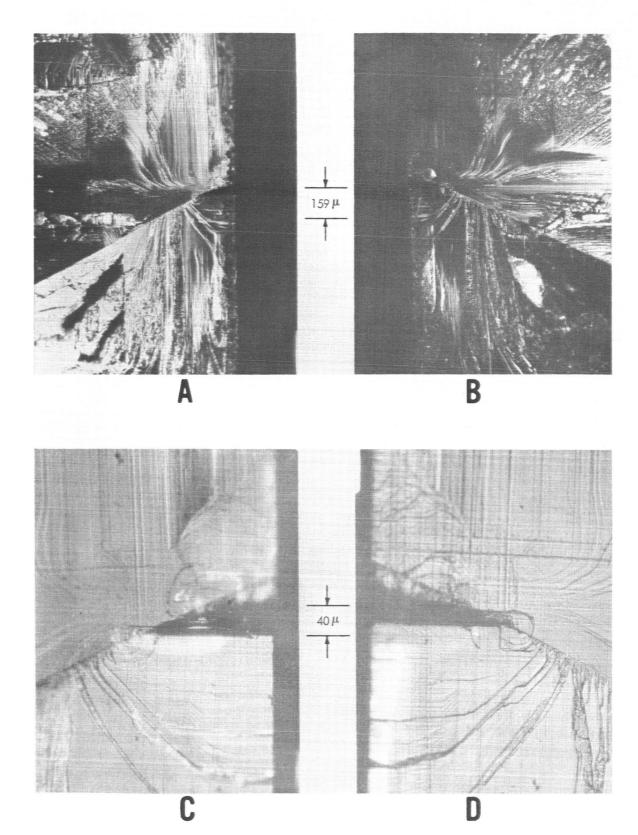


FIGURE 4 - FRACTURE SURFACE OF MgO Bi-CRYSTAL Mb-3-1 (A) & (B) Matching unetched halves, (C) & (D) Matching etched halves